

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Robert D. Harlan Examiner #: 77263 Date: 5/7/03
 Art Unit: 1713 Phone Number 306-5926 Serial Number: 09/936,902
 Mail Box and Bldg/Room Location: CP3-2A09 Results Format Preferred (circle): PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need.

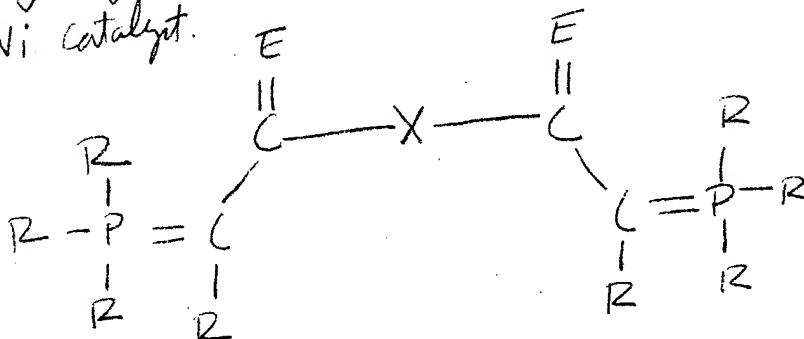
Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Method for polymerizing olefin
 Inventors (please provide full names): Tomov et al

Earliest Priority Filing Date: 09-19-01

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Please search for the following ligand as part of a Ni catalyst.



R is alkyl or phenyl

X is or $-CH_2-CH_2-$ or $-CH_2-$

E = O, S (Not much for compounds with the metal. A few hits on the organic ligand alone)

STAFF USE ONLY

| | Type of Search | Vendors and cost where applicable |
|---------------------------------------|--------------------------|-----------------------------------|
| Searcher: <u>Ed</u> | NA Sequence (#) _____ | STN <u>\$205.89</u> |
| Searcher Phone #: _____ | AA Sequence (#) _____ | Dialog _____ |
| Searcher Location: _____ | Structure (#) <u>(3)</u> | Questel/Orbit _____ |
| Date Searcher Picked Up: _____ | Bibliographic _____ | Dr.Link _____ |
| Date Completed: <u>5-8-03</u> | Litigation _____ | Lexis/Nexis _____ |
| Searcher Prep & Review Time: <u>5</u> | Fulltext _____ | Sequence Systems _____ |
| Clerical Prep Time: _____ | Patent Family _____ | WWW/Internet _____ |
| Online Time: <u>60</u> | Other _____ | Other (specify) _____ |

=> file reg

FILE 'REGISTRY' ENTERED AT 12:33:59 ON 09 MAY 2003
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FILE 'LREGISTRY' ENTERED AT 11:48:36 ON 09 MAY 2003

L1 STR
L2 STR

FILE 'REGISTRY' ENTERED AT 12:00:52 ON 09 MAY 2003

L3 0 S L1 AND L2
L4 STR L1
L5 0 S L4 AND L2
L6 STR L2
L7 0 S L4 AND L6
L8 STR L4
L9 0 S L8 AND L6
L10 0 S L8 AND L6 FUL

FILE 'BEILSTEIN' ENTERED AT 12:10:48 ON 09 MAY 2003

L11 1 S L8
L12 0 S L8 AND L6
L13 13 S L8 FUL
SAV L13 HAR902/A
L14 0 S L8 AND L6 SSS SAM SUB=L13
L15 0 S L8 AND L6 SSS FUL SUB=L13
L16 12 S L13 AND 1907-2001/PY

FILE 'REGISTRY' ENTERED AT 12:33:59 ON 09 MAY 2003

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L6 STR
M 1

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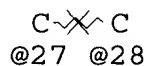
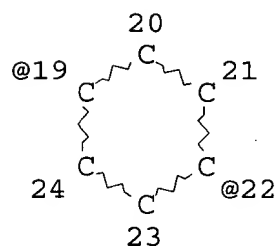
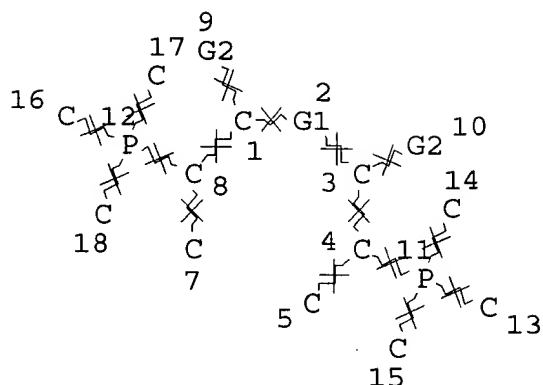
NSPEC IS RC AT 1
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 1

STEREO ATTRIBUTES: NONE

L8 STR



VAR G1=19-1 22-3/27-1 28-3/C

VAR G2=O/S

NODE ATTRIBUTES:

| | | | |
|-------|-------|----|----|
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| NSPEC | IS RC | AT | 7 |
| NSPEC | IS RC | AT | 13 |
| NSPEC | IS RC | AT | 14 |
| NSPEC | IS RC | AT | 15 |
| NSPEC | IS RC | AT | 16 |
| NSPEC | IS RC | AT | 17 |
| NSPEC | IS RC | AT | 18 |

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 25

STEREO ATTRIBUTES: NONE

L10 0 SEA FILE=REGISTRY SSS FUL L8 AND L6

100.0% PROCESSED 48921 ITERATIONS

SEARCH TIME: 00.00.01

0 ANSWERS

=> file beilstein

FILE 'BEILSTEIN' ENTERED AT 12:34:15 ON 09 MAY 2003

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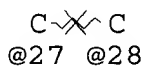
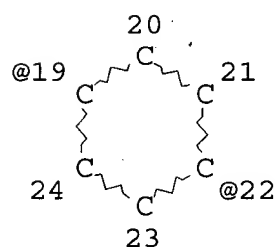
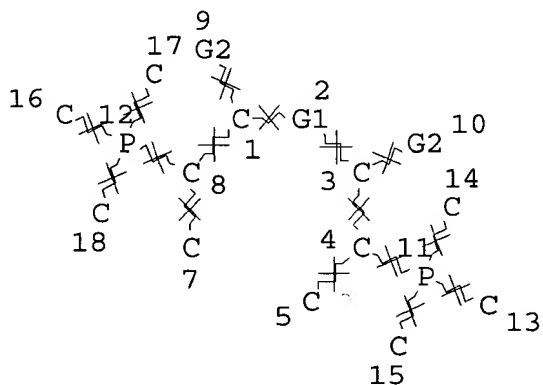
FILE RELOADED ON OCTOBER 20, 2002

FILE LAST UPDATED ON APRIL 10, 2003

FILE COVERS 1771 TO 2003.

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L8 STR



VAR G1=19-1 22-3/27-1 28-3/C

VAR G2=O/S

NODE ATTRIBUTES:

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|-------|----|----|----|----|
| NSPEC | IS | RC | AT | 5 |
| NSPEC | IS | RC | AT | 7 |
| NSPEC | IS | RC | AT | 13 |
| NSPEC | IS | RC | AT | 14 |
| NSPEC | IS | RC | AT | 15 |
| NSPEC | IS | RC | AT | 16 |
| NSPEC | IS | RC | AT | 17 |

NSPEC IS RC AT 18
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 25

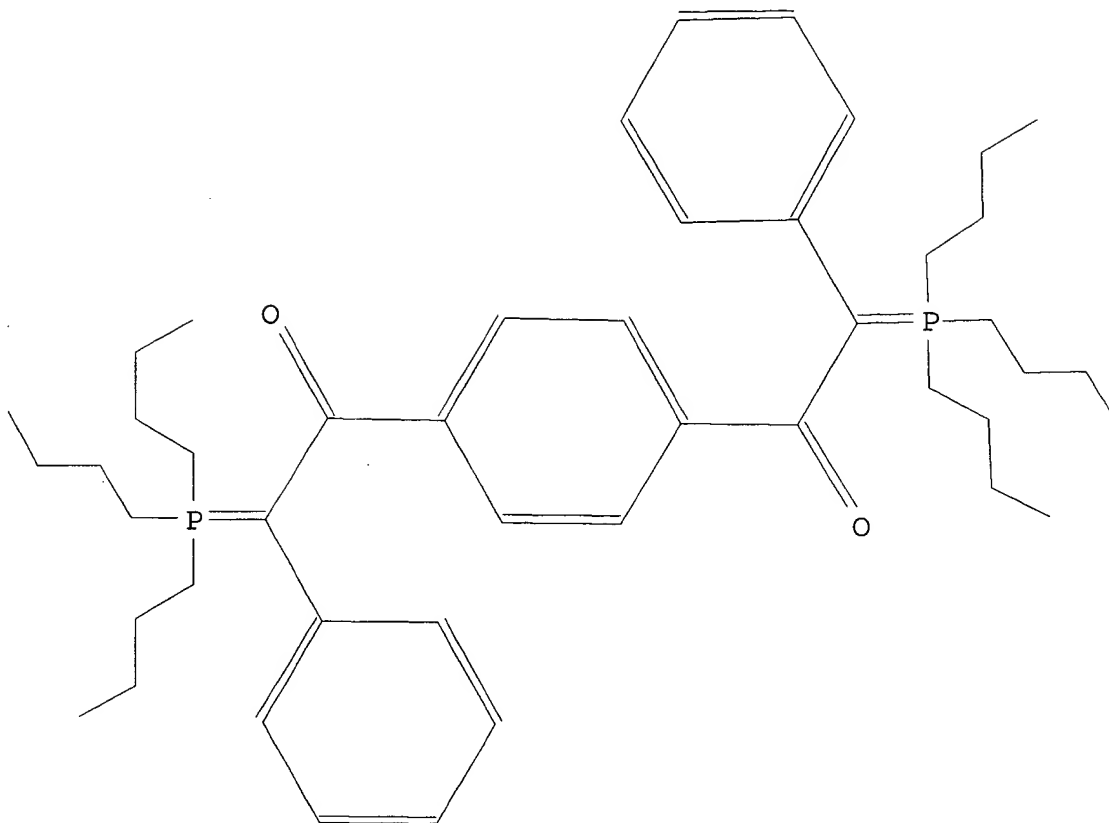
STEREO ATTRIBUTES: NONE
L13 .13 SEA FILE=BEILSTEIN SSS FUL L8

100.0% PROCESSED 1131 ITERATIONS 13 ANSWERS
SEARCH TIME: 00.00.06

=> d l16 2,4,6,8 all

L16 ANSWER 2 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

| | |
|---------------------------|--|
| Beilstein Records (BRN): | 8379815 |
| Chemical Name (CN): | 1,4-bis<(phenyl)(tributylphosphorany lidene)acetyl>benzene |
| Autonom Name (AUN): | 2-phenyl-1-<4-<phenyl-(tributyl- .lambda.5-phosphanylidene)-acetyl>- phenyl>-2-(tributyl-.lambda.5- phosphanylidene)-ethanone |
| Molec. Formula (MF): | C46 H68 O2 P2 |
| Molecular Weight (MW): | 714.99 |
| Lawson Number (LN): | 16730, 3764 |
| Compound Type (CTYPE): | isocyclic |
| Constitution ID (CONSID): | 7115632 |
| Tautomer ID (TAUTID): | 7904874 |
| Entry Date (DED): | 2000/03/08 |
| Update Date (DUPD): | 2000/03/08 |



Field Availability:

| Code | Name | Occurrence |
|--------|------------------------------|------------|
| ===== | | |
| BRN | Beilstein Records | 1 |
| CN | Chemical Name | 1 |
| AUN | Autonomname | 1 |
| MF | Molecular Formula | 1 |
| FW | Formular Weight | 1 |
| LN | Lawson Number | 2 |
| CTYPE | Compound Type | 1 |
| CONSID | Constitution ID | 1 |
| TAUTID | Tautomer ID | 1 |
| ED | Entry Date | 1 |
| UPD | Update Date | 1 |
| CPD | Crystal Property Description | 1 |
| IR | Infrared Spectrum | 1 |
| MP | Melting Point | 1 |
| NMR | Nuclear Magnetic Resonance | 3 |

This substance also occurs in Reaction Documents:

| Code | Name | Occurrence |
|-------|-------------------------------|------------|
| RX | Reaction Documents | 1 |
| RXPRO | Substance is Reaction Product | 1 |

Crystal Property Description:
CPD

(CPD): yellow

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Melting Point:

| Value (MP) (Cel) | Ref. |
|------------------------|------|
| 177 - 180 | 1 |

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Nuclear Magnetic Resonance:

NMR

| | |
|------------------------|---|
| Coupling Nuclei (.NUI) | 1H-1H |
| Solvents (.SOL): | CDCl3 |
| Frequency (.F): | 300 MHz |
| Reference(s): | 1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053 |

NMR

| | |
|--------------------|---|
| Description (.KW): | Chemical shifts |
| Nucleus (.NUC): | 1H |
| Solvents (.SOL): | CDCl3 |
| Frequency (.F): | 300 MHz |
| Reference(s): | 1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053 |

NMR

| | |
|--------------------|-----------------|
| Description (.KW): | Chemical shifts |
| Nucleus (.NUC): | 31P |
| Solvents (.SOL): | CDCl3 |
| Frequency (.F): | 32 MHz |
| Reference(s): | |

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Infrared Spectrum:

| Descript ion (.KW) | Solvent (.SOL) | Ref. |
|--------------------------|-------------------|-------|
| ===== | ===== | ===== |
| Bands | nujol | 1 |

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Reaction:

RX

Reaction ID (.ID): 5215853
Reactant BRN (.RBRN): 3776226, 607796
Reactant (.RCT): benzyl-tributyl-phosphonium;
chloride, terephthaloyl dichloride
Product BRN (.PBRN): 8379815
Product (.PRO): 2-phenyl-1-<4-<phenyl-(tributyl-
.lambda.5-phosphanylidene)-acetyl>-
phenyl>-2-(tributyl-.lambda.5-
phosphanylidene)-ethanone
No. of React. Details (.NVAR): 1

Reaction Details:

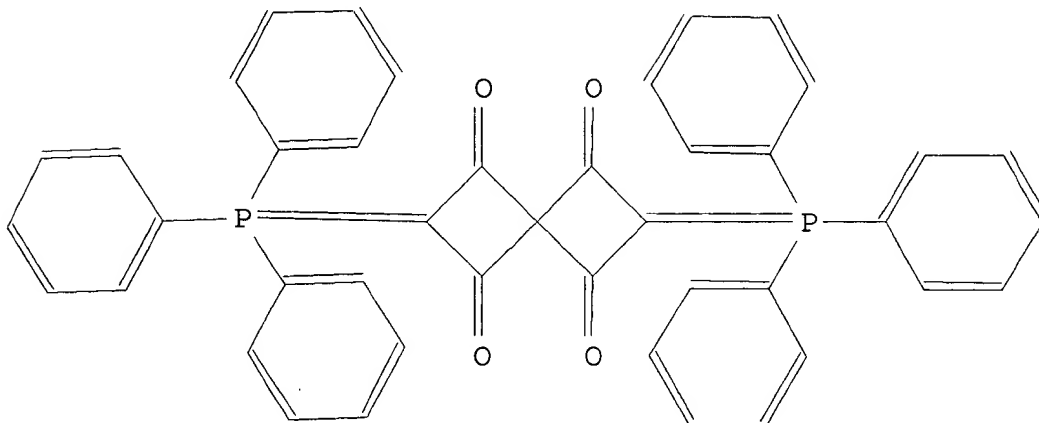
RX

Reaction RID (.RID): 5215853.1
Reaction Classification (.CL): Preparation
Yield (.YDT): 50 percent (BRN=8379815)
Reagent (.RGT): BuLi
Solvent (.SOL): tetrahydrofuran, hexane
Time (.TIM): 12 hour(s)
Reaction Type (.TYP): Acylation, transylation
Reference(s):
1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

L16 ANSWER 4 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

Beilstein Records (BRN): 7453566
Chemical Name (CN): 2,6-bis-(triphenyl-.lambda.5-
phosphanylidene)-spiro<3.3>heptane-

Autonom Name (AUN): 1,3,5,7-tetraone
 2,6-bis-(triphenyl-.lambda.5-
 phosphanylidene)-spiro<3.3>heptane-
 1,3,5,7-tetraone
 Molec. Formula (MF): C43 H30 O4 P2
 Molecular Weight (MW): 672.66
 Lawson Number (LN): 16735, 16731
 Compound Type (CTYPE): isocyclic
 Constitution ID (CONSID): 6392524
 Tautomer ID (TAUTID): 7063132
 Beilstein Citation (BSO): 6-16
 Entry Date (DED): 1996/08/09
 Update Date (DUPD): 1997/04/28



Field Availability:

| Code | Name | Occurrence |
|--------|--------------------|------------|
| BRN | Beilstein Records | 1 |
| CN | Chemical Name | 1 |
| AUN | Autonomname | 1 |
| MF | Molecular Formula | 1 |
| FW | Formular Weight | 1 |
| LN | Lawson Number | 2 |
| FS | File Segment | 1 |
| CTYPE | Compound Type | 1 |
| CONSID | Constitution ID | 1 |
| TAUTID | Tautomer ID | 1 |
| BSO | Beilstein Citation | 1 |
| ED | Entry Date | 1 |
| UPD | Update Date | 1 |
| CDEN | Density (Crystal) | 1 |
| CRYPH | Crystal Phase | 1 |

| | | |
|------|-------------------------------|---|
| CSG | Crystal Space Group | 1 |
| CSYS | Crystal System | 1 |
| GEO | Interatomic Distanc and Angle | 1 |
| IR | Infrared Spectrum | 1 |
| MP | Melting Point | 1 |
| MS | Mass Spectrum | 1 |
| NMR | Nuclear Magnetic Resonance | 4 |

This substance also occurs in Reaction Documents:

| Code | Name | Occurrence |
|-------|--------------------------------|------------|
| ===== | ===== | ===== |
| RX | Reaction Documents | 2 |
| RXREA | Substance is Reaction Reactant | 1 |
| RXPRO | Substance is Reaction Product | 1 |

Interatomic Distance and Angle:

GEO

Description (.KW): Interatomic distances and angles
 Reference(s):
 1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

Melting Point:

| Value (MP) (Cel) | Solvent (.SOL) | Ref. | Note |
|------------------------|-------------------|-------|-------|
| ===== | ===== | ===== | ===== |
| 245 - 250 | CH2Cl2 | 1 | 1 |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

Notes(s):

1. 50

Crystal Phase:

CRYPH

Description (.KW): Crystal structure determination
 Note(s) (.COM): alpha=102.2 grad, beta=90.4 grad,
 .chi.=75.4 grad, a=14.99 Angstroem,
 b=18.95 Angstroem, c=12.91
 Angstroem, n=2., Temperature: 298 K.
 Method of determination: Single
 Crystal X-ray Diffraction

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

Crystal System:

CSYS

CSYS: triclinic

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

Crystal Space Group:

CSG

CSG: C1i

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

Crystal Density:

| Value (CDEN) (g/cm**3) | Ref. |
|------------------------------|------|
| 1.29 | 1 |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

Nuclear Magnetic Resonance:

NMR

| | |
|--------------------|-----------------|
| Description (.KW): | Chemical shifts |
| Nucleus (.NUC): | 1H |
| Solvents (.SOL): | CD2Cl2 |
| Temperature (.T): | 25 Cel |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77; BABS-6009844

NMR

| | |
|--------------------|-----------------|
| Description (.KW): | Chemical shifts |
| Nucleus (.NUC): | 13C |
| Solvents (.SOL): | CD2Cl2 |
| Temperature (.T): | 25 Cel |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis,

Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1),
<1996>, 75-77; BABS-6009844

NMR

Description (.KW): Chemical shifts
Nucleus (.NUC): ³¹P
Solvents (.SOL): CD₂Cl₂
Temperature (.T): 25 Cel
Reference(s):
1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis,
Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1),
<1996>, 75-77; BABS-6009844

NMR

Description (.KW): Spin-spin coupling constants
Solvents (.SOL): CD₂Cl₂
Temperature (.T): 25 Cel
Note(s) (.COM): ³¹P-¹³C.
Reference(s):
1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis,
Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1),
<1996>, 75-77; BABS-6009844

Infrared Spectrum:

| Descript ion (.KW) | Solvent (.SOL) | Ref. | Note |
|--------------------------|-------------------|-------|-------|
| ===== | ===== | ===== | ===== |
| Bands | nujol | 1 | 1 |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo;
Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1), <1996>, 75-77;
BABS-6009844

Notes(s):

1. 1662 cm⁻¹

Mass Spectrum:

MS
Description (.KW): spectrum
Note(s) (.COM): FAB (fast atom bombardment)
Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis,
Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1),
<1996>, 75-77; BABS-6009844

Reaction:

RX

Reaction ID (.ID): 4420782
Reactant BRN (.RBRN): 1697986, 2811397

Reactant (.RCT): propadienedione,
(triphenylphosphoranylidene)ethenone
Product BRN (.PBRN): 7453566
Product (.PRO): 2,6-bis-(triphenyl-.lambda.5-
phosphanylidene)-spiro<3.3>heptane-
1,3,5,7-tetraone
No. of React. Details (.NVAR): 1

Reaction Details:

RX

Reaction RID (.RID): 4420782.1
Reaction Classification (.CL): Preparation
Yield (.YDT): 86 percent (BRN=7453566)
Solvent (.SOL): toluene
Other Conditions (.COND): Ambient temperature
Reference(s):
1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis,
Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1),
<1996>, 75-77; BABS-6009844

Reaction:

RX

Reaction ID (.ID): 4433110
Reactant BRN (.RBRN): 7453566, 505984
Reactant (.RCT): 2,6-bis-(triphenyl-.lambda.5-
phosphanylidene)-spiro<3.3>heptane-
1,3,5,7-tetraone, acetaldehyde
Product BRN (.PBRN): 7424608
Product (.PRO): 2,6-diethylidene-spiro<3.3>heptane-
1,3,5,7-tetraone
No. of React. Details (.NVAR): 1

Reaction Details:

RX

Reaction RID (.RID): 4433110.1
Reaction Classification (.CL): Preparation
Solvent (.SOL): nitromethane
Temperature (.T): 60 Cel
Reference(s):
1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis,
Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 108(1),
<1996>, 75-77; BABS-6009844

L16 ANSWER 6 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

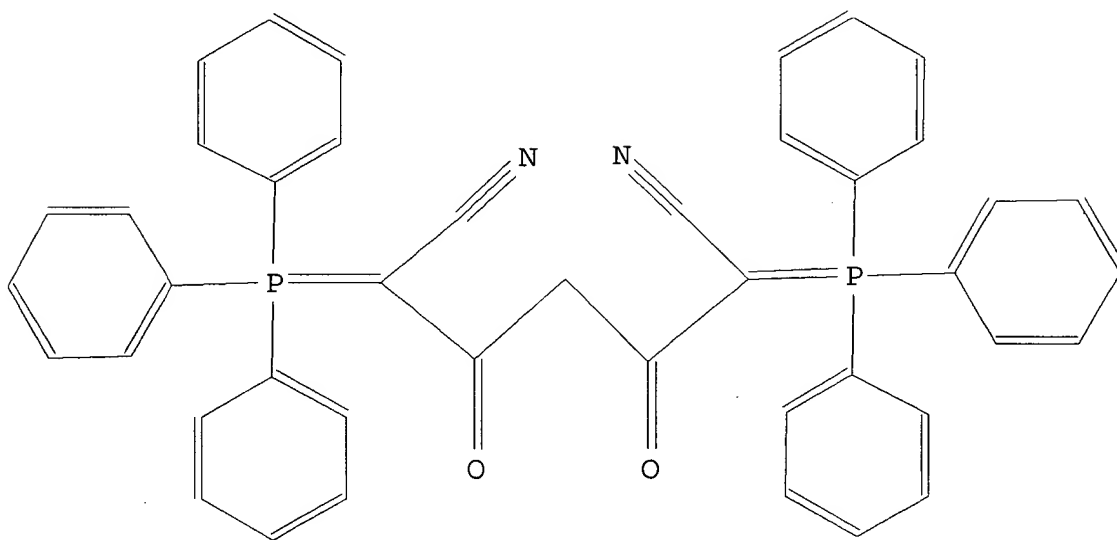
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Fragm. Molec. Formula (FMF): C43 H32 N2 O2 P2 , C7 H8
Molecular Formula (MF): 2 C43 H32 N2 O2 P2 . 3 C7 H8
Molecular Weight (MW): 670.69, 92.14
Fragment BRN (FBRN): 6854445, 635760
Lawson Number (LN): 16731, 4108, 3763

Compound Type (CTYPE): isocyclic
Constitution ID (CONSID): 5945621
Tautomer ID (TAUTID): 6526783
Beilstein Citation (BSO): 6-16
Entry Date (DED): 1994/10/31
Update Date (DUPD): 1994/10/31

CM 1

FBRN 6854445

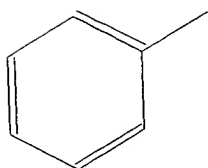
FMF C43 H32 N2 O2 P2



CM 2

FBRN 635760

FMF C7 H8



Field Availability:

Code Name

Occurrence

```
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BRN      Beilstein Records      1
FMF      Fragment Molecular Formula 2
MF       Molecular Formula      1
FW       Formular Weight        2
FBRN     Fragment BRN          2
LN       Lawson Number          3
CTYPE    Compound Type         1
CONSID   Constitution ID       1
TAUTID   Tautomer ID           1
BSO      Beilstein Citation     1
ED       Entry Date             1
UPD      Update Date            1
CDEN     Density (Crystal)      1
CRYPH    Crystal Phase         1
CSG      Crystal Space Group    1
CSYS     Crystal System         1
GEO      Interatomic Distanc and Angle 1
```

Interatomic Distance and Angle:

GEO

Description (.KW): Interatomic distances and angles
Reference(s):
1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Crystal Phase:

CRYPH

Description (.KW): Crystal structure determination
Note(s) (.COM): alpha=77.8 grad, beta=86.5 grad, .chi.=62.6 grad, a=10.2 Angstroem, b=10.53 Angstroem, c=23.88 Angstroem, n=2., Temperature: 298 K. Method of determination: Single Crystal X-ray Diffraction

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Crystal System:

CSYS

CSYS: triclinic

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Crystal Space Group:

CSG

CSG: C1i

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Crystal Density:

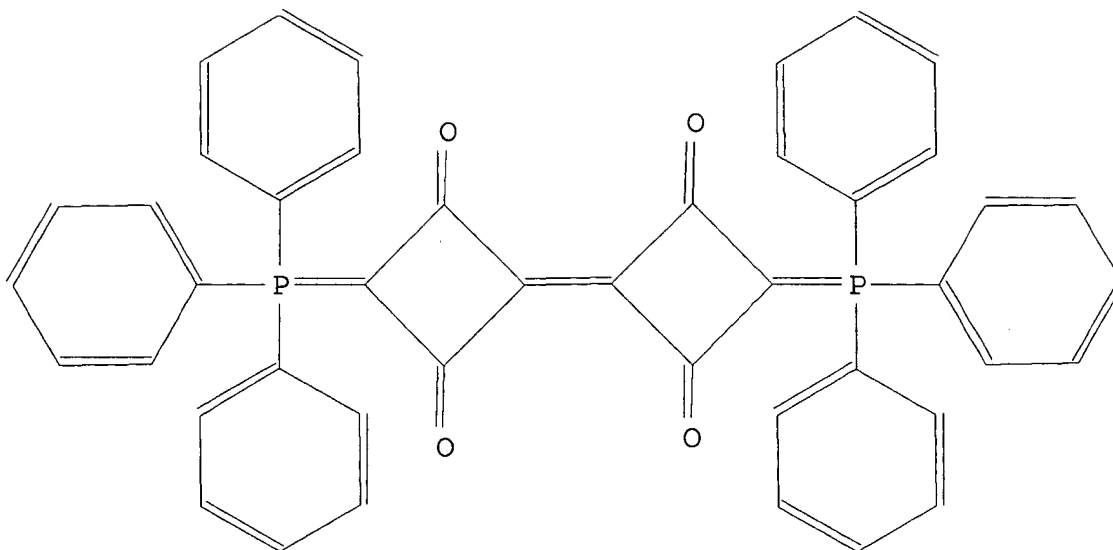
| Value (CDEN) (g/cm**3) | Ref. |
|------------------------------|------|
| 1.21 | 1 |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

L16 ANSWER 8 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

| | |
|---------------------------|---|
| Beilstein Records (BRN): | 6675719 |
| Chemical Name (CN): | 3,3'-bis-(triphenyl-.lambda.5-phosphanylidene)-bicyclobutylidene-2,4,2',4'-tetraone |
| Autonom Name (AUN): | 3,3'-bis-(triphenyl-.lambda.5-phosphanylidene)-bicyclobutylidene-2,4,2',4'-tetraone |
| Molec. Formula (MF): | C44 H30 O4 P2 |
| Molecular Weight (MW): | 684.67 |
| Lawson Number (LN): | 16731, 16728 |
| Compound Type (CTYPE): | isocyclic |
| Constitution ID (CONSID): | 5787582 |
| Tautomer ID (TAUTID): | 6323323 |
| Beilstein Citation (BSO): | 6-16 |
| Entry Date (DED): | 1994/07/18 |
| Update Date (DUPD): | 1995/05/11 |



Field Availability:

| Code | Name | Occurrence |
|--------|-------------------------------|------------|
| BRN | Beilstein Records | 1 |
| CN | Chemical Name | 1 |
| AUN | Autonomname | 1 |
| MF | Molecular Formula | 1 |
| FW | Formular Weight | 1 |
| LN | Lawson Number | 2 |
| FS | File Segment | 1 |
| CTYPE | Compound Type | 1 |
| CONSID | Constitution ID | 1 |
| TAUTID | Tautomer ID | 1 |
| BSO | Beilstein Citation | 1 |
| ED | Entry Date | 1 |
| UPD | Update Date | 1 |
| CDEN | Density (Crystal) | 1 |
| CRYPH | Crystal Phase | 1 |
| CSG | Crystal Space Group | 1 |
| CSYS | Crystal System | 1 |
| GEO | Interatomic Distanc and Angle | 1 |
| IR | Infrared Spectrum | 1 |
| MP | Melting Point | 1 |
| NMR | Nuclear Magnetic Resonance | 1 |

This substance also occurs in Reaction Documents:

| Code | Name | Occurrence |
|------|--------------------|------------|
| RX | Reaction Documents | 3 |

| | | |
|-------|--------------------------------|---|
| RXREA | Substance is Reaction Reactant | 1 |
| RXPRO | Substance is Reaction Product | 2 |

Interatomic Distance and Angle:

GEO

Description (.KW): Interatomic distances and angles

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Melting Point:

| Value (MP) (Cel) | Ref. | Note |
|------------------------|-------|-------|
| ===== | ===== | ===== |
| | 1 | 1 |

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Notes(s):

1. Details: sintered at: 300 C - 360 C

Crystal Phase:

CRYPH

Description (.KW):

Note(s) (.COM):

Crystal structure determination
alpha=81.6 grad, beta=86.9 grad,
.chi.=88 grad, a=9.28 Angstroem,
b=9.31 Angstroem, c=9.95 Angstroem,
n=1., Temperature: 22 C. Method of
determination: X-ray Diffraction

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Crystal System:

CSYS

CSYS:

triclinic

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Crystal Space Group:

CSG

CSG:

C1i

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Crystal Density:

| Value (CDEN) (g/cm**3) | Ref. |
|------------------------------|------|
| 1.338 | 1 |

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Nuclear Magnetic Resonance:

NMR

| | |
|--------------------|-----------------|
| Description (.KW): | Chemical shifts |
| Nucleus (.NUC): | 31P |
| Solvents (.SOL): | CDCl3 |
| Temperature (.T): | 25 Cel |
| Reference(s): | |

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Infrared Spectrum:

| Descript ion (.KW) | Solvent (.SOL) | Ref. | Note |
|--------------------------|-------------------|------|------|
| Bands | KBr | 1 | 1 |

Reference(s):

1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette, Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784; BABS-5852222

Notes(s):

1. 1650 - 1640 cm**(-1)

Reaction:

| | |
|-----------------------|-------------------------------|
| RX | |
| Reaction ID (.ID): | 2171987 |
| Reactant BRN (.RBRN): | 2795610 |
| Reactant (.RCT): | 2,4-bis-(triphenyl-.lambda.5- |

phosphanylidene)-cyclobutane-1,3-dione
Product BRN (.PBRN): 6675719
Product (.PRO): 3,3'-bis-(triphenyl-.lambda.5-phosphanylidene)-bicyclobutylidene-2,4,2',4'-tetraone
No. of React. Details (.NVAR): 1

Reaction Details:

RX

Reaction RID (.RID): 2171987.1
Reaction Classification (.CL): Preparation
Yield (.YDT): 98 percent (BRN=6675719)
Reagent (.RGT): N-p-tolylsulfonyl(phenyl)oxaziridine
Solvent (.SOL): diethyl ether
Reference(s):
1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette,
Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784;
BABS-5852222

Reaction:

RX

Reaction ID (.ID): 2171985
Reactant BRN (.RBRN): 2795610, 6657405
Reactant (.RCT): 2,4-bis-(triphenyl-.lambda.5-phosphanylidene)-cyclobutane-1,3-dione, 4-(triphenyl-.lambda.5-phosphanylidene)-cyclobutane-1,2,3-trione
Product BRN (.PBRN): 6675719
Product (.PRO): 3,3'-bis-(triphenyl-.lambda.5-phosphanylidene)-bicyclobutylidene-2,4,2',4'-tetraone
No. of React. Details (.NVAR): 1

Reaction Details:

RX

Reaction RID (.RID): 2171985.1
Reaction Classification (.CL): Preparation
Reference(s):
1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette,
Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784;
BABS-5852222

Reaction:

RX

Reaction ID (.ID): 3312996
Reactant BRN (.RBRN): 6675719, 605285
Reactant (.RCT): 3,3'-bis-(triphenyl-.lambda.5-phosphanylidene)-bicyclobutylidene-2,4,2',4'-tetraone, 2,3-dimethyl-buta-1,3-diene

Product BRN (.PBRN): 6678270
Product (.PRO): 10,11-dimethyl-2,7-bis-(triphenyl-
.lambda.5-phosphanylidene)-
dispiro<3.0.3.4>dodec-10-ene-1,3,6,8-
tetraone
No. of React. Details (.NVAR): 1

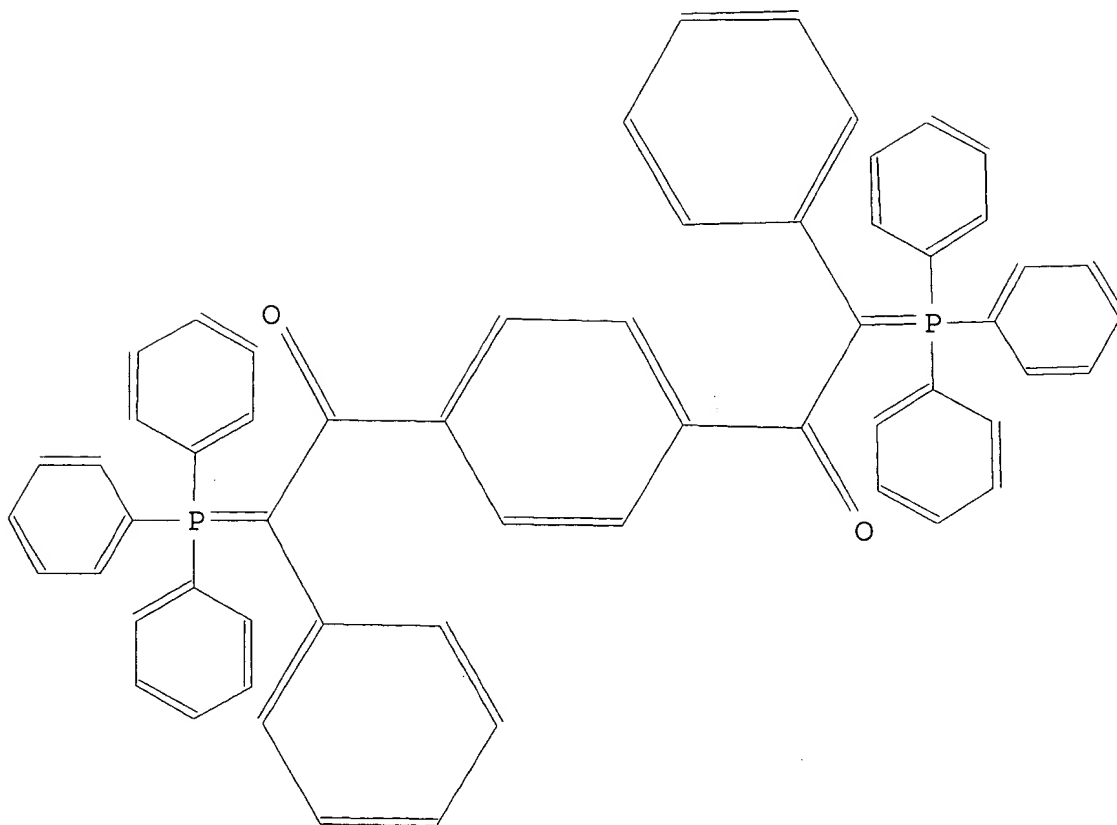
Reaction Details:

RX
Reaction RID (.RID): 3312996.1
Reaction Classification (.CL): Preparation
Yield (.YDT): 58 percent (BRN=6678270)
Solvent (.SOL): nitrobenzene
Time (.TIM): 24 hour(s)
Other Conditions (.COND): Heating
Reference(s):
1. Bestmann, Hans Juergen; Fuerst, Thomas G.; Schier, Annette,
Angew.Chem., CODEN: ANCEAD, 105(12), <1993>, 1783-1784;
BABS-5852222

=> d 116 3,5,9 all

L16 ANSWER 3 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

Beilstein Records (BRN): 8379169
Chemical Name (CN): 1,4-bis<(phenyl)(triphenylphosphoran
ylidene)acetyl>benzene
Autonom Name (AUN): 2-phenyl-1-<4-<(phenyl-(triphenyl-
.lambda.5-phosphanylidene)-acetyl>-
phenyl>-2-(triphenyl-.lambda.5-
phosphanylidene)-ethanone
Molec. Formula (MF): C58 H44 O2 P2
Molecular Weight (MW): 834.93
Lawson Number (LN): 16731, 16730
Compound Type (CTYPE): isocyclic
Constitution ID (CONSID): 7115121
Tautomer ID (TAUTID): 7889594
Entry Date (DED): 2000/03/08
Update Date (DUPD): 2000/03/08



Field Availability:

| Code | Name | Occurrence |
|--------|------------------------------|------------|
| ===== | ===== | ===== |
| BRN | Beilstein Records | 1 |
| CN | Chemical Name | 1 |
| AUN | Autonomname | 1 |
| MF | Molecular Formula | 1 |
| FW | Formular Weight | 1 |
| LN | Lawson Number | 2 |
| FS | File Segment | 1 |
| CTYPE | Compound Type | 1 |
| CONSID | Constitution ID | 1 |
| TAUTID | Tautomer ID | 1 |
| ED | Entry Date | 1 |
| UPD | Update Date | 1 |
| CPD | Crystal Property Description | 1 |
| IR | Infrared Spectrum | 1 |
| MP | Melting Point | 1 |
| MS | Mass Spectrum | 1 |
| NMR | Nuclear Magnetic Resonance | 4 |

This substance also occurs in Reaction Documents:

| Code | Name | Occurrence |
|-------|-------------------------------|------------|
| RX | Reaction Documents | 1 |
| RXPRO | Substance is Reaction Product | 1 |

Crystal Property Description:

CPD

(CPD): yellow

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Melting Point:

| Value (MP) (Cel) | Ref. |
|------------------------|------|
| 270 - 274 | 1 |

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Nuclear Magnetic Resonance:

NMR

Coupling Nuclei (.NUI) 31P-13C
Solvents (.SOL): CDCl3
Frequency (.F): 75 MHz

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

NMR

Description (.KW): Chemical shifts
Nucleus (.NUC): 31P
Solvents (.SOL): CDCl3
Frequency (.F): 32 MHz

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

NMR

Description (.KW): Chemical shifts
Nucleus (.NUC): 1H
Solvents (.SOL): CDCl3

Frequency (.F): 300 MHz

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

NMR

Description (.KW): Chemical shifts

Nucleus (.NUC): ¹³CSolvents (.SOL): CDCl₃

Frequency (.F): 75 MHz

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Infrared Spectrum:

| Descript ion (.KW) | Solvent (.SOL) | Ref. |
|--------------------------|-------------------|------|
| Bands | nujol | 1 |

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Mass Spectrum:

MS

Description (.KW): spectrum, electron impact (EI)

Reference(s):

1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

Reaction:

RX

Reaction ID (.ID): 5215852

Reactant BRN (.RBRN): 3599868, 607796

Reactant (.RCT): benzyl-triphenyl-phosphonium;
chloride, terephthaloyl dichloride

Product BRN (.PBRN): 8379169

Product (.PRO): 2-phenyl-1-(4-(phenyl-(triphenyl-
.lambda.5-phosphanylidene)-acetyl)-
phenyl)-2-(triphenyl-.lambda.5-
phosphanylidene)-ethanone

No. of React. Details (.NVAR): 1

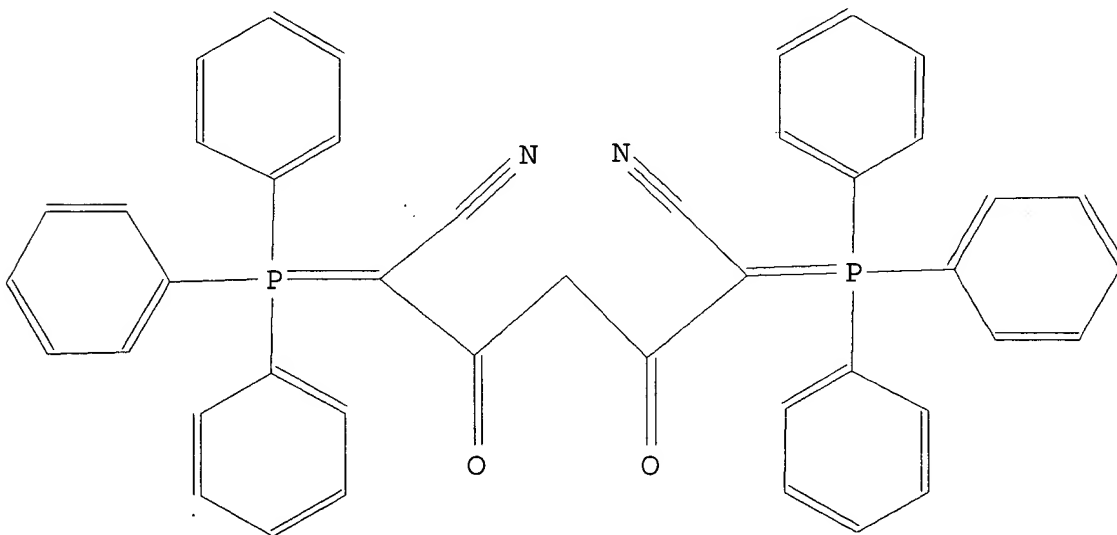
Reaction Details:

RX

Reaction RID (.RID): 5215852.1
Reaction Classification (.CL): Preparation
Yield (.YDT): 60 percent (BRN=8379169)
Reagent (.RGT): BuLi
Solvent (.SOL): tetrahydrofuran, hexane
Time (.TIM): 12 hour(s)
Reaction Type (.TYP): Acylation, transylation
Reference(s):
1. Aitken, R. Alan; Drysdale, Martin J.; Hill, Lawrence; Lumbard, Keith W.; MacCallum, James R.; Seth, Shirley, Tetrahedron, CODEN: TETRAB, 55(36), <1999>, 11039 - 11050; BABS-6182053

L16 ANSWER 5 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

Beilstein Records (BRN): 6854445
Chemical Name (CN): 1,3-bis(cyanomethylenetriphenylphosphorane)propane-1,3-dione
Autonom Name (AUN): 3,5-dioxo-2,6-bis-(triphenyl-
.lambda.5-phosphanylidene)-
heptanedinitrile
Molec. Formula (MF): C43 H32 N2 O2 P2
Molecular Weight (MW): 670.69
Lawson Number (LN): 16731, 3763
Compound Type (CTYPE): isocyclic
Constitution ID (CONSID): 5939736
Tautomer ID (TAUTID): 6516581
Beilstein Citation (BSO): 6-16
Entry Date (DED): 1994/10/31
Update Date (DUPD): 1996/04/26



Field Availability:

| Code | Name | Occurrence |
|--------|----------------------------|------------|
| BRN | Beilstein Records | 1 |
| CN | Chemical Name | 1 |
| AUN | Autonomname | 1 |
| MF | Molecular Formula | 1 |
| FW | Formular Weight | 1 |
| LN | Lawson Number | 2 |
| FS | File Segment | 1 |
| CTYPE | Compound Type | 1 |
| CONSID | Constitution ID | 1 |
| TAUTID | Tautomer ID | 1 |
| BSO | Beilstein Citation | 1 |
| ED | Entry Date | 1 |
| UPD | Update Date | 1 |
| IR | Infrared Spectrum | 1 |
| MP | Melting Point | 1 |
| MS | Mass Spectrum | 2 |
| NMR | Nuclear Magnetic Resonance | 4 |

This substance also occurs in Reaction Documents:

| Code | Name | Occurrence |
|-------|-------------------------------|------------|
| RX | Reaction Documents | 1 |
| RXPRO | Substance is Reaction Product | 1 |

Melting Point:

| Value (MP) (Cel) | Ref. | Note |
|------------------------|------|------|
| 243 - 244 | 1 | 1 |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Notes(s):

1. 50

Nuclear Magnetic Resonance:

NMR

Description (.KW):

Chemical shifts

Nucleus (.NUC): 1H
 Solvents (.SOL): CD2Cl2
 Temperature (.T): 25 Cel
 Reference(s):
 1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

NMR

Description (.KW): Chemical shifts
 Nucleus (.NUC): 13C
 Solvents (.SOL): CD2Cl2
 Temperature (.T): 25 Cel
 Reference(s):
 1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

NMR

Description (.KW): Chemical shifts
 Nucleus (.NUC): 31P
 Solvents (.SOL): CDCl3
 Temperature (.T): 25 Cel
 Reference(s):
 1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

NMR

Description (.KW): Spin-spin coupling constants
 Solvents (.SOL): CD2Cl2
 Temperature (.T): 25 Cel
 Note(s) (.COM): 1H-13C, 31P-13C.
 Reference(s):
 1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Infrared Spectrum:

| Descript ion (.KW) | Solvent (.SOL) | Ref. | Note |
|--------------------------|-------------------|------|------|
| Bands | nujol | 1 | 1 |

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

Notes(s):

1. 2175 - 1565 cm**(-1)

Mass Spectrum:

MS
Description (.KW): spectrum
Note(s) (.COM): FAB (fast atom bombardment)

Reference(s):

1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

MS

Description (.KW): fragmentation pattern
Note(s) (.COM): FAB (fast atom bombardment),
metastable ions

Reference(s):

1. Seraglia, Roberta; Traldi, Pietro; Bertani, Roberta; Facchin, Giacomo; Pandolfo, Luciano, Org.Mass Spectrom., CODEN: ORMSBG, 29(11), <1994>, 619-624; BABS-5944502

Reaction:

RX
Reaction ID (.ID): 3775143
Reactant BRN (.RBRN): 750218, 1697986
Reactant (.RCT): (triphenyl-.lambda.5-phosphanylidene)-acetonitrile,
propadienedione
Product BRN (.PBRN): 6854445
Product (.PRO): 3,5-dioxo-2,6-bis-(triphenyl-.lambda.5-phosphanylidene)-
heptanedinitrile
No. of React. Details (.NVAR): 1

Reaction Details:

RX
Reaction RID (.RID): 3775143.1
Reaction Classification (.CL): Preparation
Yield (.YDT): 85 percent (BRN=6854445)
Solvent (.SOL): 1,2-dichloro-ethane
Other Conditions (.COND): Ambient temperature
Reference(s):
1. Pandolfo, Luciano; Facchin, Giacomo; Bertani, Roberta; Ganis, Paolo; Valle, Giovanni, Angew.Chem., CODEN: ANCEAD, 106(5), <1994>, 586-588; BABS-5894226

L16 ANSWER 9 OF 12 BEILSTEIN COPYRIGHT 2003 BEILSTEIN CDS MDL

Beilstein Records (BRN): 6459041
Chemical Name (CN): 2,7-bis-(triphenyl-.lambda.5-phosphanylidene)-octane-3,6-dione
Autonom Name (AUN): 2,7-bis-(triphenyl-.lambda.5-phosphanylidene)-octane-3,6-dione

Reference(s):

1. Sanehi, Ram; Bansal, R. K.; Mehrotra, R. C., Indian J.Chem.Sect.A, CODEN: IJCADU, 24(5), <1985>, 398-402; BABS-5793414

Notes(s):

1. 1540 cm**(-1)

Reaction:

RX

| | |
|--------------------------------|---|
| Reaction ID (.ID): | 3879158 |
| Reactant BRN (.RBRN): | 1773914, 958776 |
| Reactant (.RCT): | 1,4-dithio-succinic acid S,S'-diethyl ester, ethylidene-triphenyl-.lambda.5- phosphane |
| Product BRN (.PBRN): | 6459041 |
| Product (.PRO): | 2,7-bis-(triphenyl-.lambda.5- phosphanylidene)-octane-3,6-dione |
| No. of React. Details (.NVAR): | 1 |

Reaction Details:

RX

| | |
|--------------------------------|--|
| Reaction RID (.RID): | 3879158.1 |
| Reaction Classification (.CL): | Preparation |
| Yield (.YDT): | 63 percent (BRN=6459041) |
| Solvent (.SOL): | toluene |
| Other Conditions (.COND): | 1.) ambient temp., 5 h, 2.) reflux, 3 h |

Reference(s):

1. Sanehi, Ram; Bansal, R. K.; Mehrotra, R. C., Indian J.Chem.Sect.A, CODEN: IJCADU, 24(5), <1985>, 398-402; BABS-5793414

| | L # | Hits | Search Text | DBs |
|---|-----|------|--|-------|
| 1 | L1 | 279 | harlan.xa. | USPAT |
| 2 | L2 | 8 | ((526/160) or (526/161) or (526/170) or (526/171) or (526/172) or (526/943) or (502/152) or (502/155)).CCLS.) and 20030513.pd. | USPAT |
| 3 | L3 | 4788 | ((502/155) or (502/167) or (502/152) or (526/160) or (526/161) or (526/170) or (526/171) or (526/172) or (526/943)).CCLS. | USPAT |
| 4 | L4 | 70 | 3 and binuclear | USPAT |
| 5 | L5 | 40 | 3 and ylide | USPAT |
| 6 | L6 | 0 | 3 and ylide and nickle | USPAT |
| 7 | L7 | 23 | 3 and ylide and nickel | USPAT |
| 8 | L8 | 2 | 3 and ylide and nickel and binuclear | USPAT |
| 9 | L9 | 1 | ("4691036").PN. | USPAT |



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